VO2 Experiment

Aug 20th ~25th. 2010

Jong Woo Kim

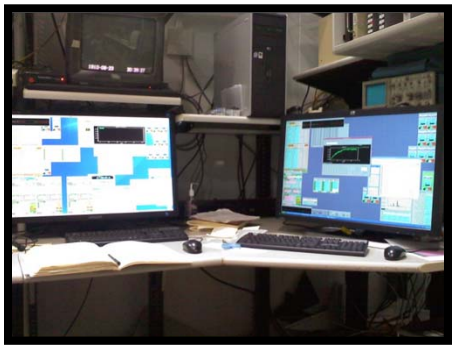
Contents

Shpyrko Research Group

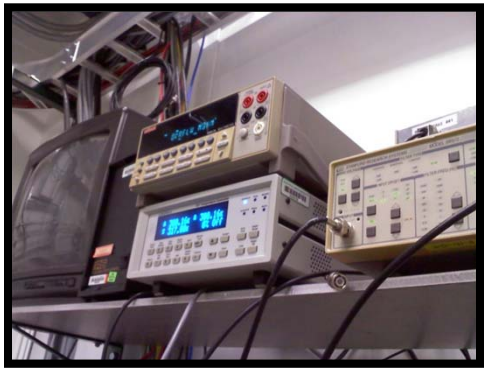
Introduction

Shpyrko Research Group

- Objective
  - X-ray induced Metal-Insulator Transition in VO2 Film
- Members
  - Sebastian, Moses, Jong Woo, Oleg Shpyrko (PI)
- Facilities



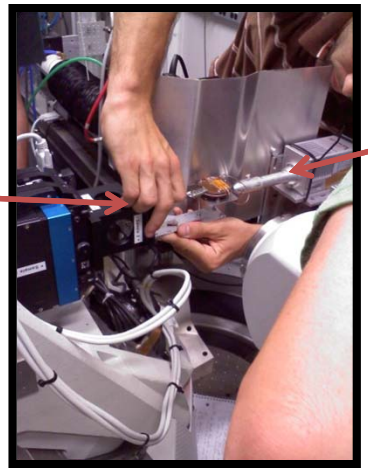
S/W : EPICS

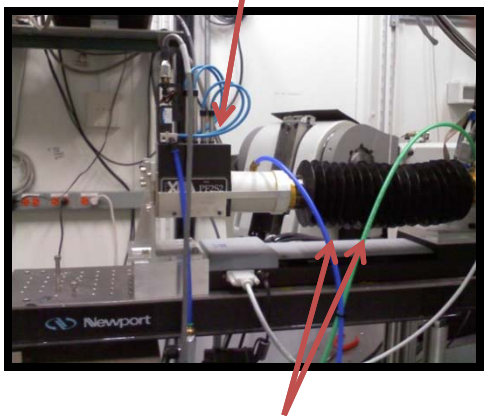


Lakeshore 331 temperature controller

Introduction

- Facilities





Introduction

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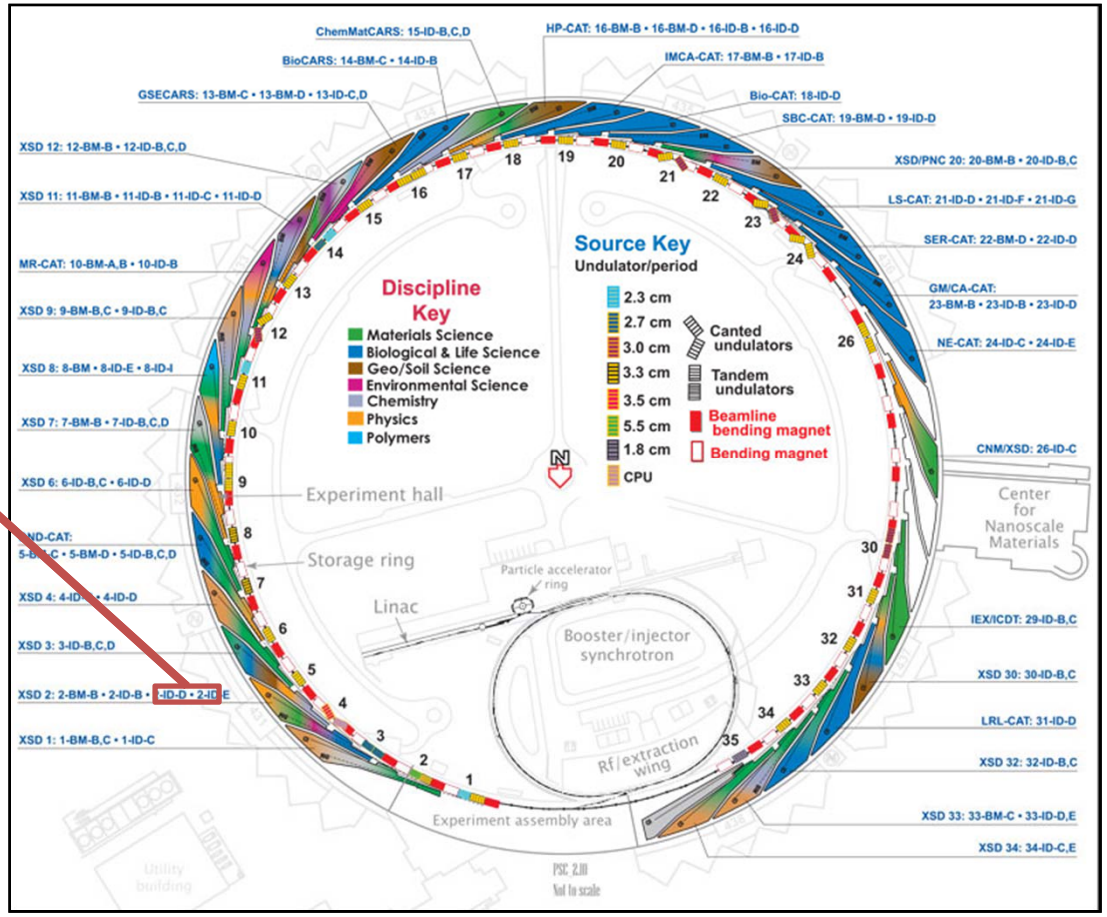
- Beamline : 2-ID-D

2-ID-D

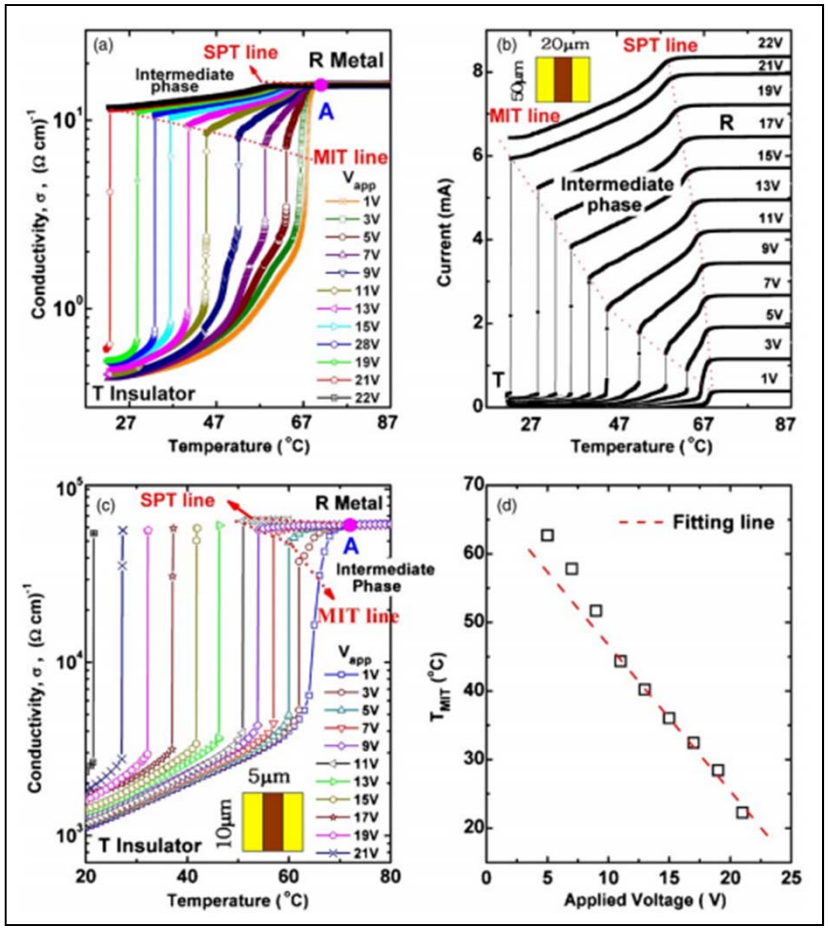


Background

- Metal-Insulator Transition(MIT)
- T_{MIT} can be modified by the followings
  - X-ray
  - Voltage
  - Stress, doping, electric field, magnetic field

Background

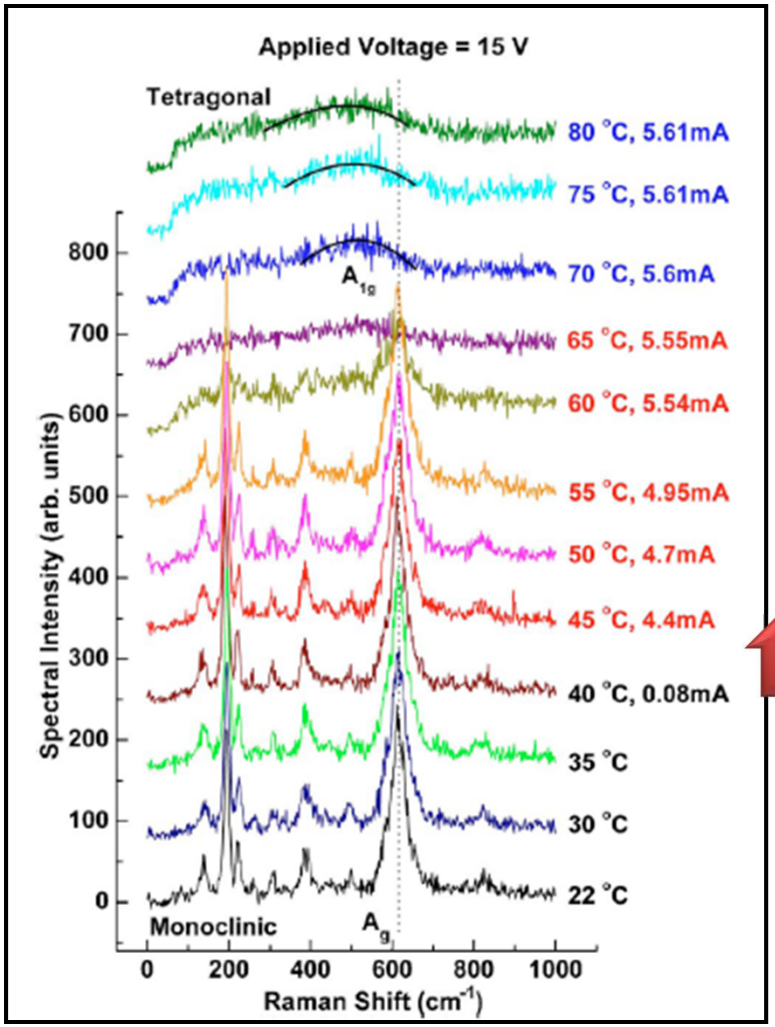
- Voltage induced MIT (Kim et al.)



Kim et al., Appl. Phys. Lett., 90, 23515 (2007)

Background

- Voltage induced MIT (Kim et al.)

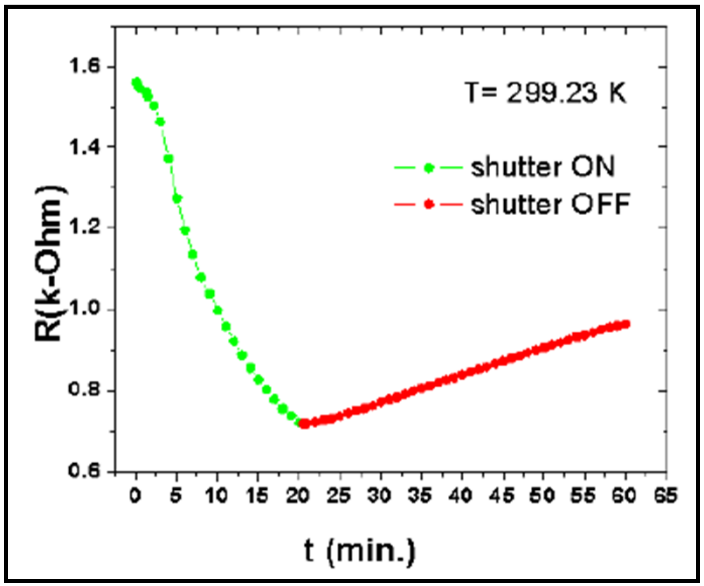


MIT

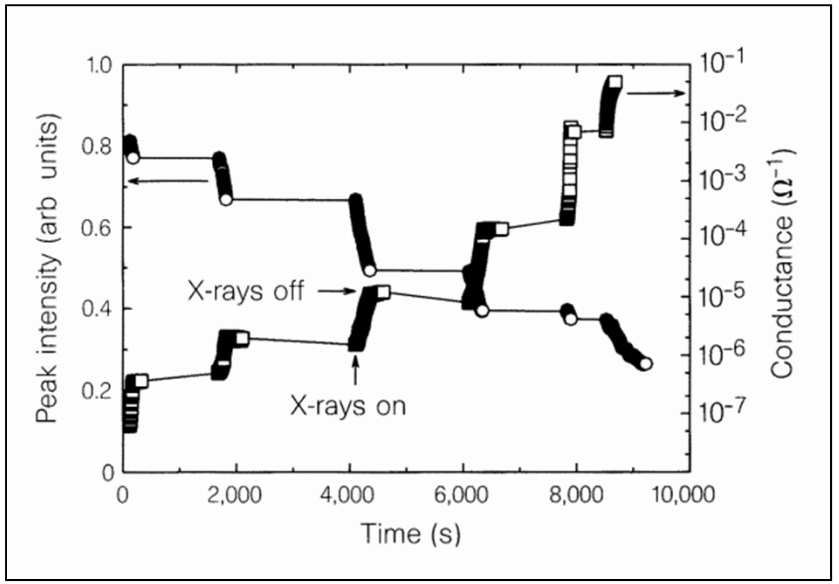
Kim et al., Appl. Phys. Lett., 90, 23515 (2007)

Background

- X-ray induced MIT (Previous experiment, Kiryukhin et al.)
  - Photo-induced & field-induced metallic phases are identical
  - X-ray induced MIT is caused by X-ray photoelectrons and secondary electrons generated in collisions, Not photo-induced oxygen diffusion neither oxygen-diffusion mechanism



Oleg group, previous experiment(2009)



Kiryukhin et al. Nature, 386, 813 (1997)

Experiment

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Day		Action	Purpose	Result	Remark
1st	1	Scan Au Fluor.	Locate Au contacts		
2nd	2	T ↗ (RT→330K)			
	3	Measure R w/XR @332.5K	Choose device	Device 4	
	4	T ↗↘(332.5→352.6K→319.9K)			
	5	V↗↘ (0→3→0V) @310K	V-induced	Metalic until V off	
3rd	6	Apply 3V @RT	V-induced	ΔR=7kΩ	
	7	V ↘↗ (3→2→3) @ RT	Joule-heating	R non-recovered	
	8	XR on @3V, RT	XR-V-induced	Went metalic	
	9	T ↗(RT→355K) @ 0, 1 and 3V	V-induced	V↑ →Transition↓	
	10	V ↘↗ (3→0→3)w/ XR off @RT	Joule-heating	R non-recovered	
	11	XR on/off @3V, RT	V-induced	Trans. Found (17kΩ→115Ω)	
4th	12	XR on w/ CCD @RT ?V	Locate Au contacts		
	13	T ↗↘ (RT→400→RT)	R back on	R returned (to 30kΩ)	
	14	XR on @RT, 0V	XR-induced	R reduced, (to 28kΩ)	
	15	XR on @RT, 2V	XR-induced under V	R reduced, (to 27.8kΩ)	
	16	XR on/off @RT, 2V	XR-induced under V	R reduced slowly	
	17	V ↗↘ @RT			
5th	18	T ↗↘(RT→400→RT)	R back on	Return to 30kΩ	
	19	XR on/off @RT, 3V	XR-induced under V		
	20	T ↗ “tail” area w/ XRD	Structural Phase Trans.	To be analyzed	
	21	T ↘ ↗(RT 345K) V↗ on D1	Max. allowable V	18V	
	22	T ↗↘ (345→400→RT)	R back on	R still low	
	23	Apply 1V, XR on/off	XR-induced trans.		
	24	T ↗ (RT→ 400K)@4V	V-induced	V↑ →Transition↓	
	25	T ↗ (RT→400K)& V ↗ (0→5.2V)			

Results

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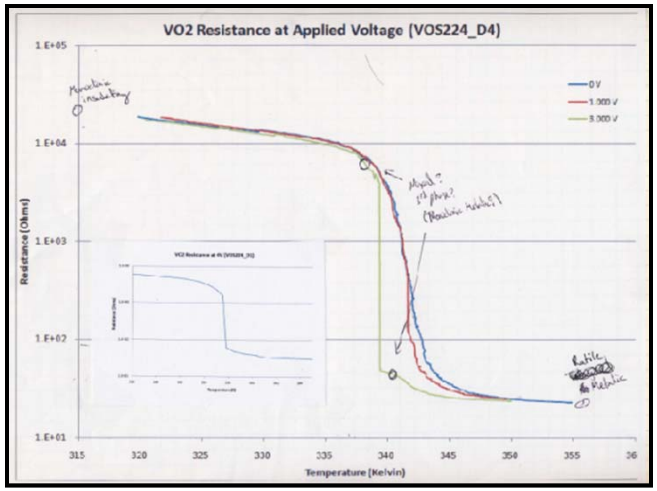
- Voltage effect
  - T_{MIT} decreases with the increasing V (#9, 25)



  - Voltage-induced transition occurs at room temperature :

    @300K, R reduced by 7kΩ @3V, but less than 2kΩ @0V or 2V (#6,14,15)

- X-Ray effect
  - T_{MIT} decreases with X-ray on, and faster when with V (#8,14,15,16,17,23)

Safety

- It is impossible to emphasize the importance of safety too much
  - Don’t rush, move slowly

Entrance process

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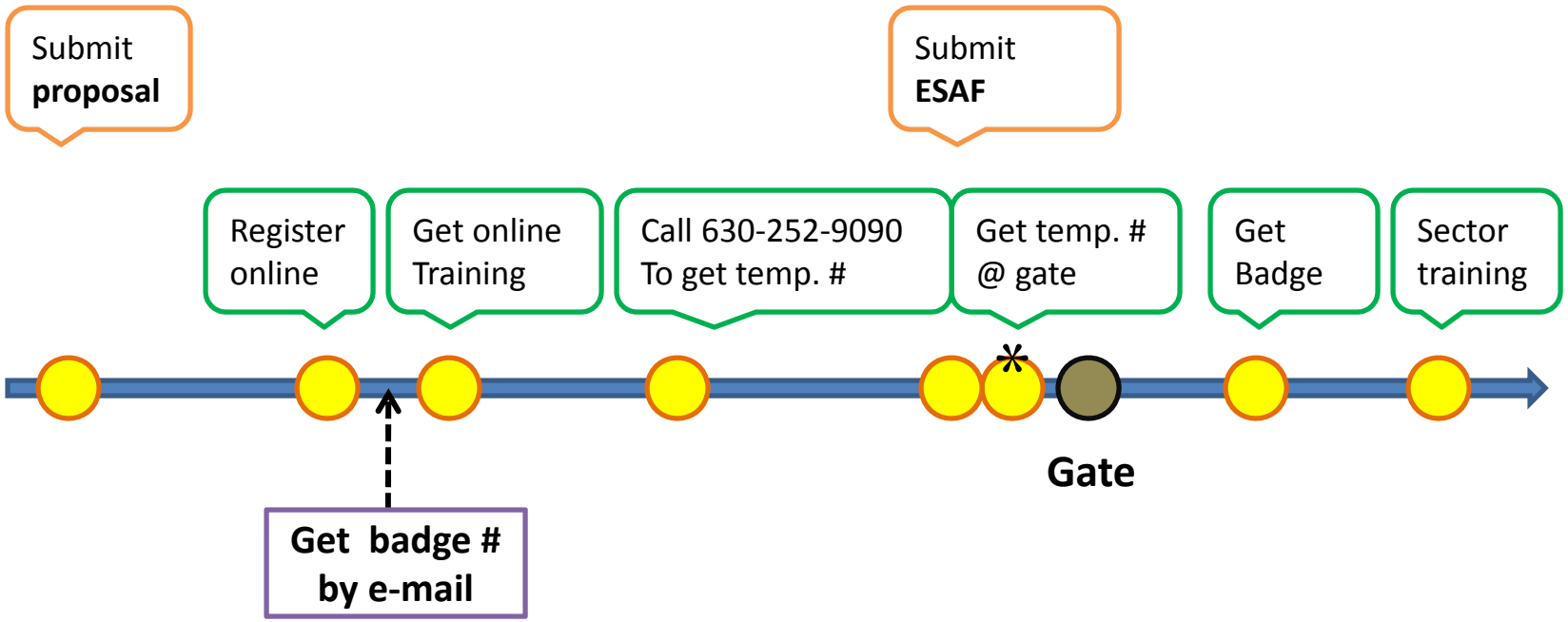

Orange : required everytime

Green : for the first visitor

* : ID required

Reference

- Kiryukhin et al. Nature, **386, 813** (1997)
- H.T Kim et al Appl. Phys. Lett., **90, 23515** (2007)

Appendix

Appendix : Experiment Details

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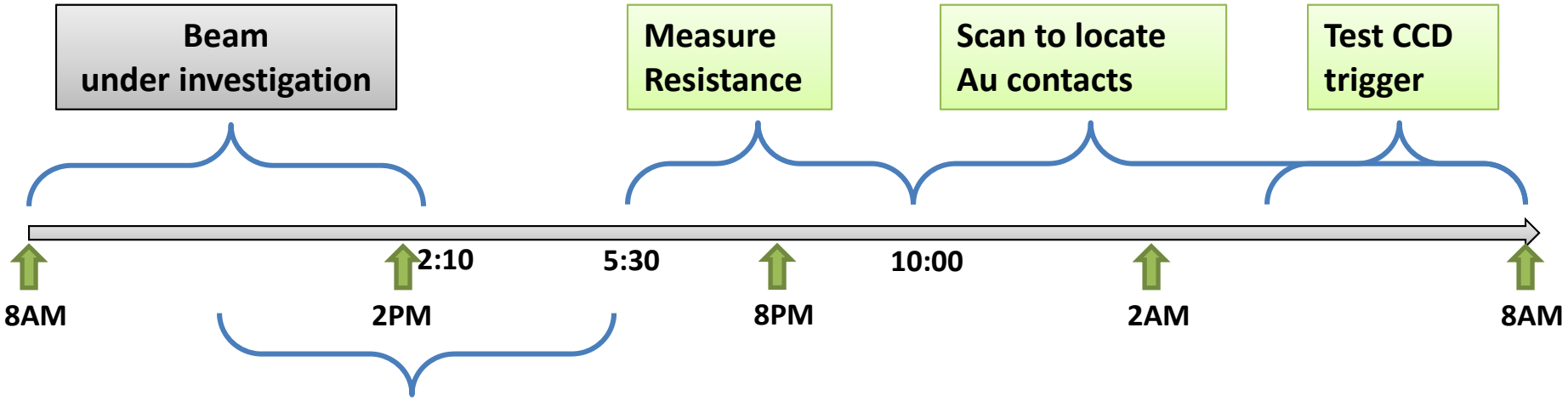
1st Day



[Sample]
-.To attach Thermocouple detector on sample
-.To identify cables/pins

[Trouble Shooting]
-.Thermocouple : K-type detector → Move outside
-.Multi-meter : Wire short found → Split & Soldering

Appendix : Experiment Details

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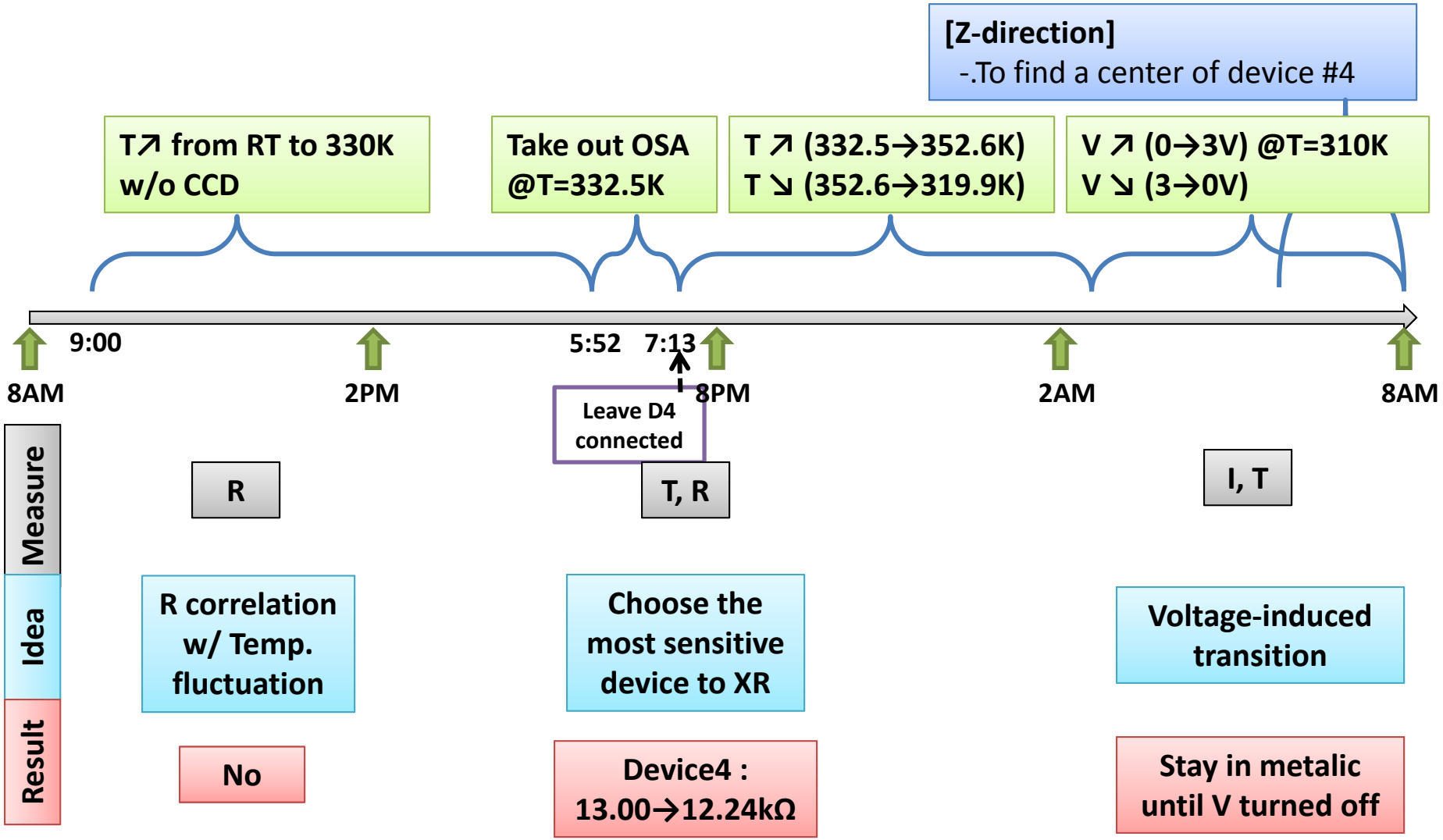
2nd Day



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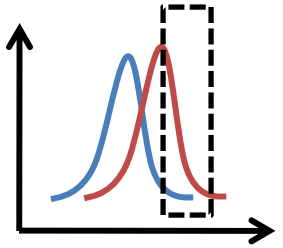
3rd Day



V ↘ ↗ (3→2→3) @ RT

Apply 3V on D4 @ RT

XR on @3V, RT

T ↗(RT→355K) @ 0, 1 and 3V

V ↘ ↗ (3→0→3) w/ XR off @RT

XR on/off @3V, RT

12:00 1:11

8AM 2PM 8PM 2AM 8AM

Measure	I	I	I	R	I	I, XRD
Idea	V-induced transition	Power effect	XR-V-induced transition	V-induced transition comparison	Power effect	V-driven Transition @RT
Result	7kΩ ↓ @ 3V	R non-recovered	Went metalic	V↑ →Transition↓	R non-recovered	Transition found 17kΩ→115Ω

Appendix : Experiment Details

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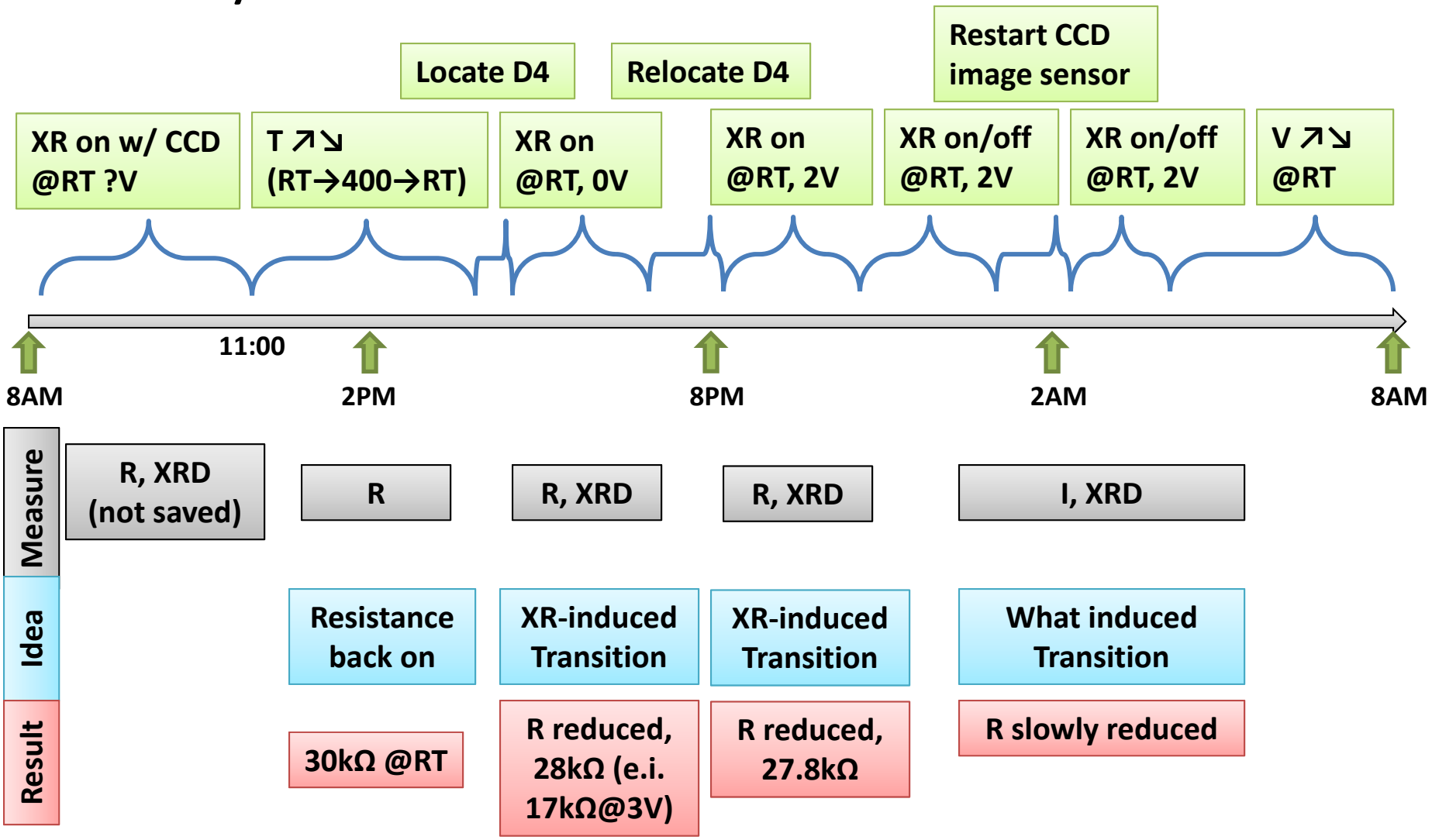
4th Day



Appendix : Experiment Details

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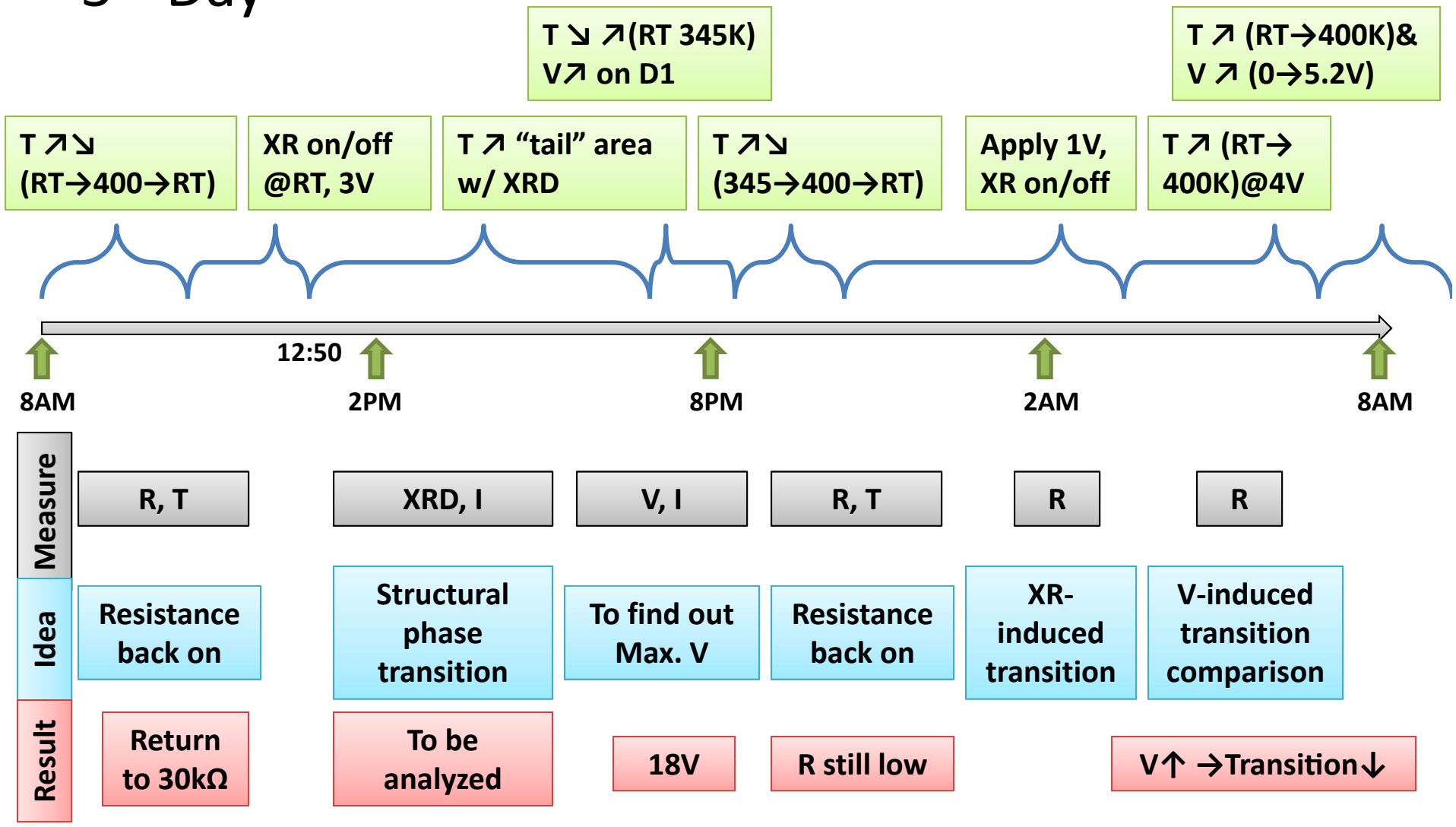
5th Day



Appendix : Experiment Data

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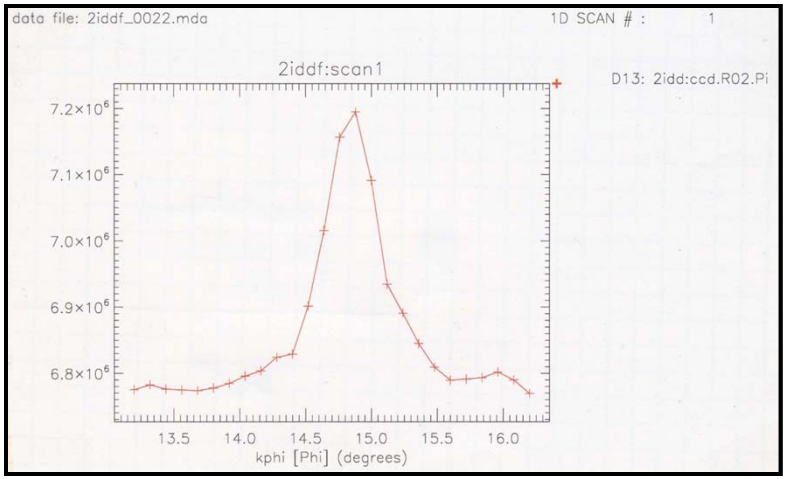
[Beam Information]

E=10.1KeV	ID Gap=10.16keV	ZP : 86.24mm

Title	Value
IC US(XRF)	280k/sec @200nA/V
IC US(XRD)	107k/sec @100nA/V
IC DS(XRD)	128k/sec @500nA/V

[Scan #1] XRD



Appendix : Experiment Data

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[Resistance]

Connection	Resistance
1A-1B	35.6kΩ
2A-2B	56.2kΩ
3A-3B	70.6kΩ
4A-4B	43.5kΩ
5A-5B	51.3kΩ

Connection	Resistance
1A-2A	35.5kΩ
2A-3A	43.7kΩ
3A-4A	36.0kΩ
4A-5A	41.5kΩ

Connection	Resistance
1B-2B	54.8kΩ
2B-3B	46.5kΩ
3B-4B	30.0kΩ
4B-5B	43.3kΩ

Connection	Resistance
1A-2B	89.9kΩ
2A-1B	97.0kΩ
2A-3B	73.5kΩ
3A-2B	72.9kΩ

Connection	Resistance
3A-4B	63.6kΩ
4A-3B	59.4kΩ
4A-5B	90.9kΩ
5A-4B	43.7kΩ

Appendix : Experiment Data

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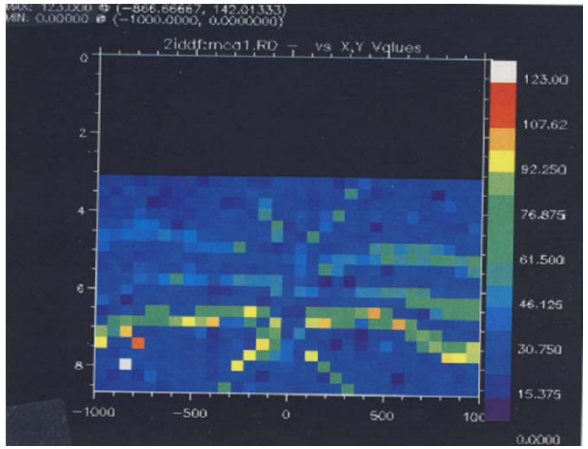
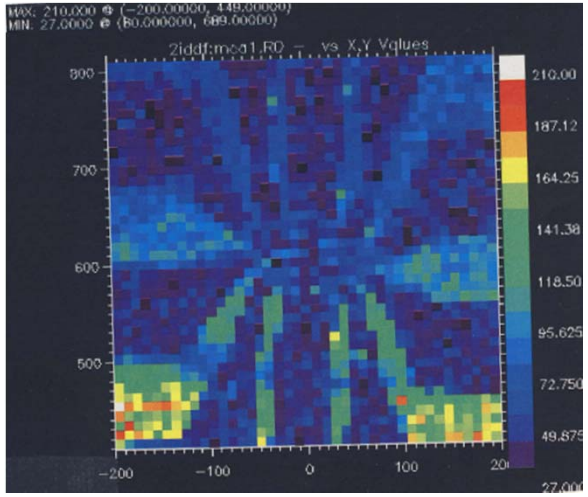
[Scan #24] Au Fluor.

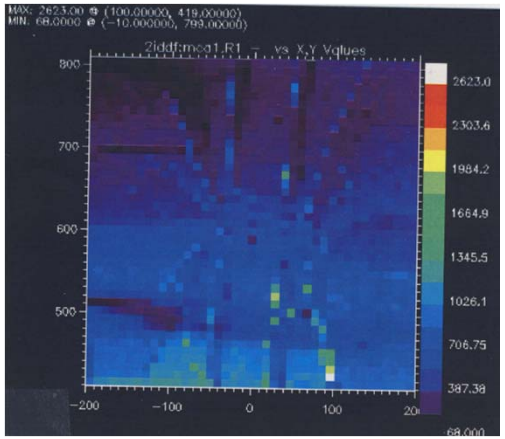


Zoom in

[Scan #25] Au Fluor.



[Scan #25] V Fluor.



Both Au & V Fluorescence Seem to reduce When going +Z (motor move upstream/ Scanning sample down stream)

[Scan #26]

Test using temp controller as dummy motor zoom in 100um X 100um scan

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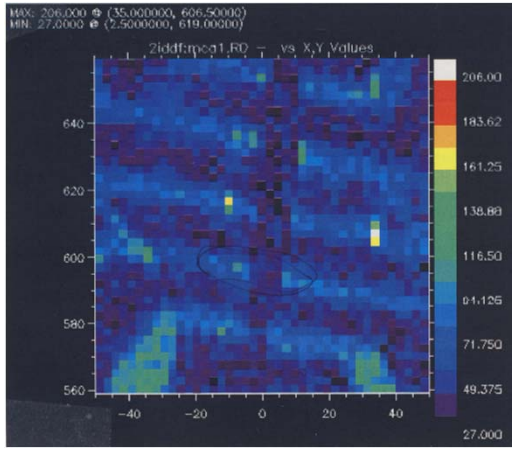
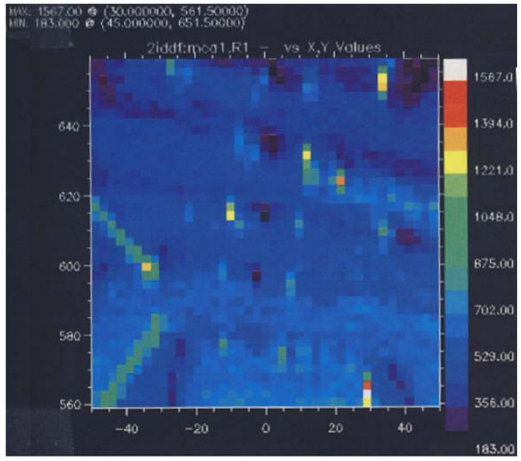
[Scan #27] Au Fluor.



⇦ D1
⇦ D2
⇦ D3
⇦ D4
⇦ D5

X=6.0 Y=652; Sample slightly rotated

[Scan #27] V Fluor.

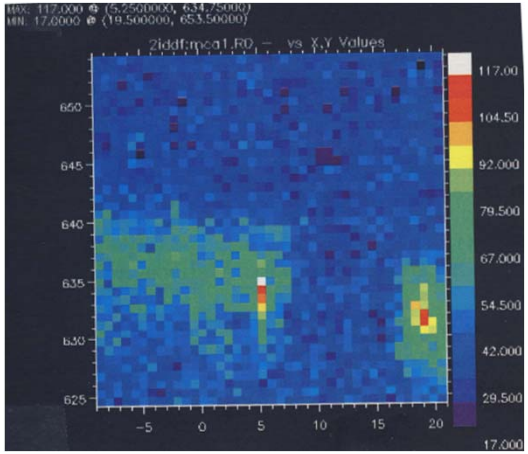


R=36.5kΩ, T=300.0K Center on D1 & 30um X30um scan

[Scan #28]

Aborted : Forgot to open shutter

[Scan #29] Au Fluor.



So far, resistance fluctuation
correspond to temperature fluctuation

Appendix : Experiment Data

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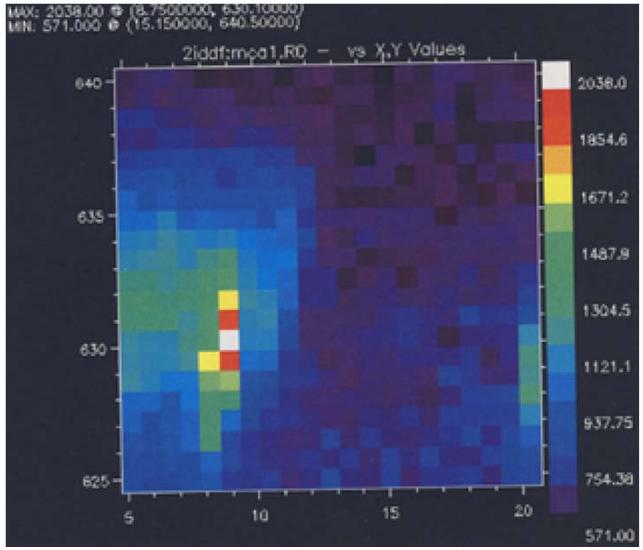
[Scan #30 & 31]

Test CCD trigger & Fluor. time is parallel to CCD time

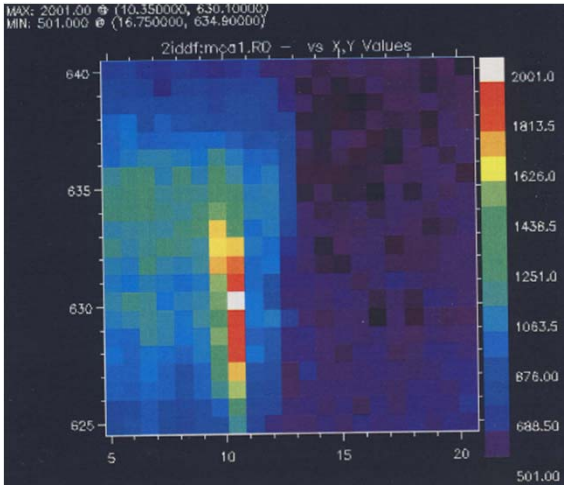
[Scan #32]

Image device #1 Loop device using temp. controller as dummy CCD & Fluor. Expos. time = 20 sec
16um X 16 um, T=300.2K

[Scan #32] Au Fluor.



[Scan #33] Au Fluor.



[Scan #34]

Changing Temp to 320K. Not collecting CCD data - Aborted (Forgot to change line & preset time from 20sec)

[Scan #35]

40um X 40um. Live time : 1sec
First two lines are of scan are blank because the shutter was closed
Bumped to T up to 330K

[Scan #36]

100um X 100um. T~330K. Drift

Appendix : Experiment Data

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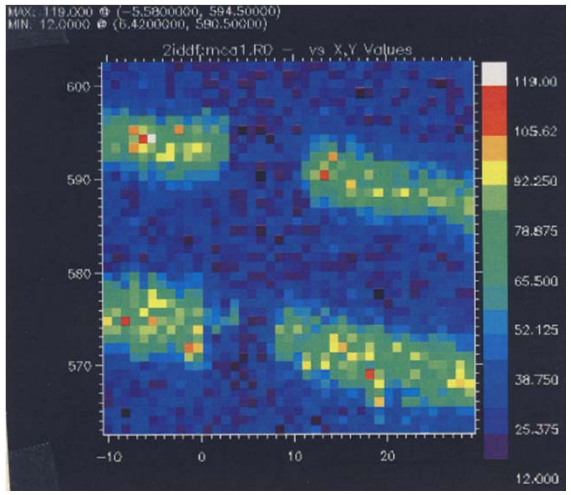
[Scan #36]

100um X 100um. T~330K
Drift – sample x went from 12 to 9.42
Sample z from 632 to 582

[Scan #37] Au Fluor.

40um X 40um. 41 points. T=330.2K
R=12.59 kΩ



[Scan #38~40] Test

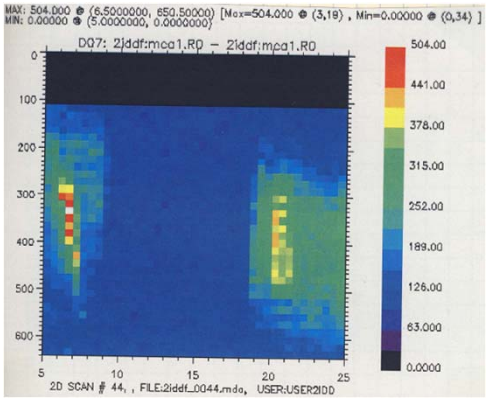
[Scan #41] 1D scan on right contacts to find top one

[Scan #42] 1D scan along top contact (Z=651, -20<X<30)

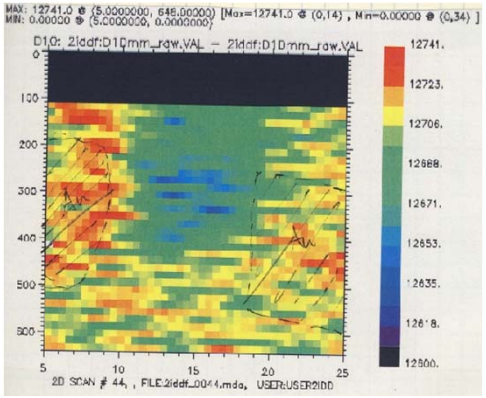
[Scan #43] Do #42 again

[Scan #44] 2D scan centered in top gap (X=15, Z=651)
20umX20um

[Scan #44] Au Fluor.



[Scan #44] Resistance



Large change does not correlate with temperature fluctuations
Check sample correction R=12.9kΩ
Temp 332.5K always staying below when approaching

Appendix : Experiment Data

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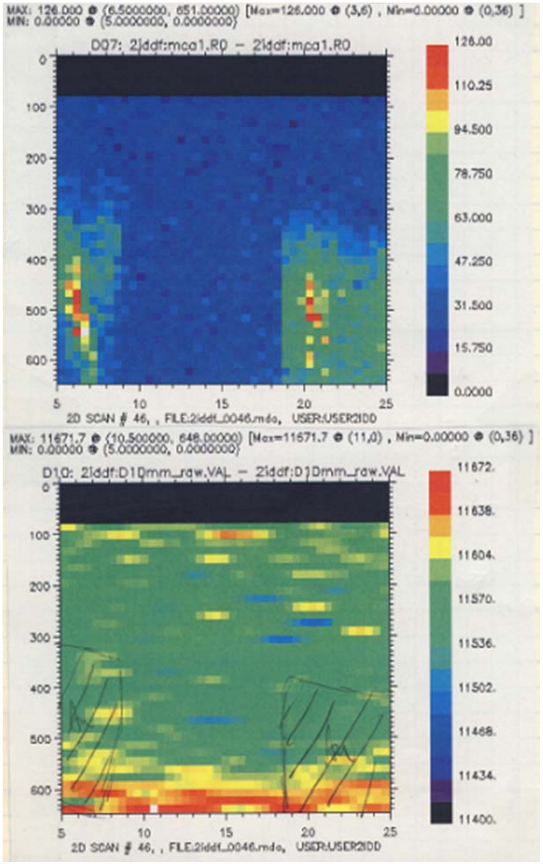
[Scan #45] Aborted

[Scan #46] Same as #44 with Flour. Real time set 50
R=11.6kΩ



Resistance correlated to temperature
No correlated to X-ray exposure b/w contacts

[Resistance of device @T=332.5]

Device	Resistance
D1	11.54kΩ
D2	13.55kΩ
D3	20.45kΩ
D4	13.00kΩ

[Scan #47, #48]
Dummy scan to record temp. & resistance

Device	Resistance
D1	11.55kΩ
D2	13.47kΩ
D3	20.45kΩ
D4	12.24kΩ

[Scan #49]
Dummy scan 1001 points to record temp & resistance while illuminating sample without OSA

[Scan #50, #51] Realign OSA X, Y

[Scan #52, #53]
Sample Z along right electrodes to find 4th device

Appendix : Experiment Data

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[Scan #61]
2D scan over device 4
R=12.27kΩ T=332.5K. Beam jumped 4/5 though

[Scan #63]
Heat through transition 332.0K → 342.6K

[Scan #64]
Same as #63 w/ 342.6K → 352.6K

[Scan #65]
Temp. sweep 320K → 355K. 2°/min outside transition, down to 0.2 °/min
Use OSA Y to collect